

What is claimed is:

(AN)  
5/30/63

~~CLAIMS~~

1. A method of obtaining selected pectin fractions having successively increasing setting times, the method comprising subjecting a starting material containing high-esterified

5 pectin to a first treatment cycle comprising a step of extracting the starting material with an aqueous medium at an acidic pH under conditions where only part of the pectin content is extracted, separating the pectin extract from the treated starting material and recovering the pectin from the 10 extract to obtain a first pectin fraction, followed by at least one further treatment cycle whereby the treated starting material extracted in the preceding cycle is treated to obtain a second and optionally one or more further pectin fractions, the pH of the extraction medium in each of the 15 second and further cycles being lower than in the immediately preceding treatment cycle.

2. A method according to claim 1 wherein the pectin-containing starting material is a pectin-containing material which has been subjected to a pre-treatment.

20 3. A method according to claim 1 wherein the pH of the aqueous medium is in the range of 1 to 4.

4. A method according to claim 1 wherein the pectin-containing material is derived from a native vegetable material in a fresh or dried state.

25 5. A method according to claim 1 wherein the pectin-containing material is the solid extraction residue from the preceding extraction step.

6. A method according to claim 1 wherein the extraction is carried out at a temperature in the range of from 40°C to 30 100°C for a period of time of from 1 to 20 hours.

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7. A method according to claim 1 wherein the extraction mixture has a content of pectin-containing material dry matter which is in the range of from 1% to 5% by weight, preferably in the range of from 2% to 4% by weight.

5 8. A method according to claim 1 wherein the aqueous solution contains an added water soluble salt of calcium, and/or aluminium in an amount which corresponds to a metal ion concentration in the range of from 10 mmol to 40 mmol per litre extraction liquid.

10 9. A method according to claim 1 wherein the pectin is recovered from the extract by precipitation with a water-miscible organic solvent in which pectin is substantially insoluble, separating the precipitated pectin from the liquid and drying the separated pectin.

15 10. A method according to claim 1 wherein the pectin is recovered from the extracts by adjusting the pH of the extract to a level in the range of 2 to 2.5, adding a strongly acidic cation exchange[r] resin in salt form to raise the pH to a level in the range of 2.5 to 3.5, stirring 20 the mixture at ambient temperature for 4 to 8 hours, separating the liquid from the ion exchange resin, precipitating the pectin by addition of a water-miscible organic solvent, separating the precipitated pectin from the liquid and drying the separated pectin.

25 11. A method according to claim 2 wherein the pre-treatment comprises heating a suspension of the pectin-containing material at a temperature of from 60°C to 80°C in a substantially homogeneous solvent mixture comprising water and at least one water-miscible organic solvent in which pectin is 30 substantially insoluble to which an acid is added to maintain a fixed pH of the suspension within the range of from 1 to 3 during the treatment and separating the pre-treated pectin-containing material.

12. A method according to claim 11 wherein the weight ratio between solvent and water in the pre-treatment mixture is from 40:60 to 80:20, the amount of water being the sum of added water and water present in the pectin containing material prior to the heat treatment.

13. A method according to claim 11 wherein the pectin-containing material is heat treated for a period of from 2 to 6 hours, preferably from 3 to 4 hours.

14. A method according to claim 11 wherein the pre-treated pectin-containing material is subjected to at least one washing step.

15. A method according to claim 11 wherein the pre-treated pectin-containing material is dried to obtain a dry matter content in the material of at least 80% by weight.

16. A method according to claim 15 wherein the pre-treated pectin-containing material is dried at a temperature in the range of from ambient temperature to 100°C for a period being at the most 36 hours.

17. A selected pectin fraction obtainable by the method of any of claims 1-16, the fraction having a degree of esterification which is at least 50% and a setting time which is the range of 0 to 100 sec, 101 to 200 sec, 201 to 300 sec or in excess of 300 sec, the setting time being determined by the method of Joseph and Bair (Food Technology, 1949, 18, 18-22) in terms of the time required for obtaining complete gelling of a hot standardized pectin-sugar-water solution at pH 2.1 to 2.5 when cooled at constant temperature of 30°C.

18. Use of a pectin fraction according to claim 17 in the preparation of a food product.

19. Use according to claim 18 wherein the food product is an acidified milk product having a pH 3.5 to 4.5.

20. Use according to claim 18 wherein the food product is a preserve.

21. A method of stabilizing an acidified milk product comprising adding to the milk product an amount of a pectin fraction as defined in claim 17, the addition of the fraction resulting in an improvement of the milk product, the improvement is selected from the group consisting of at least 10% reduction in viscosity, at least 10% smaller particles and at least 10% less sediment, as compared to the addition of the same amount of a bulk-extracted pectin product.

22. A method of according to claim 21 wherein the improvement is selected from the group consisting of at least 2 times reduction in viscosity, at least 2 times smaller particles and at least 2 times less sediment, as compared to the addition of the same amount of a bulk-extracted pectin product.

23. A method of obtaining a deesterified pectin fraction, comprising subjecting a selected pectin fraction obtainable by the method of any of claims 1-16 and having a degree of esterification which is 50% or higher, to at least one de-  
20 esterification treatment step comprising reacting the high-esterified pectin fraction with a deesterifying agent to obtain a pectin fraction having a degree of esterification (DE) which is reduced by at least 5% relative to that of the high-esterified pectin fraction and a degree of amidation  
25 (DA) which is in the range of 0-25.

24. A method according to claim 23 wherein the deesterified pectin fraction having a DE which is reduced by at least 5% relative to that of the high-esterified pectin fraction is used as a starting material in a further step of deesterification.

25. A method according to claim 23 wherein the resulting deesterified pectin fraction has a DE which is at the most 70%

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such as less than 60%, including less than 50% such as less than 45%.

26. A method according to claim 25 wherein the deesterified pectin fraction has a DE which is in the range of 20-45.

5 27. A method according to claim 23 wherein the deesterifying agent is selected from the group consisting of an acid and ammonia.

28. A method according to claim 27 wherein the deesterification comprises heat treatment of the selected pectin fraction 10 in an aqueous solution or suspension in the presence of an acid to give a pH of at the most 1.

29. A method according to claim 27, wherein the deesterification occurs in a reaction mixture having a content of pectin dry matter which is in the range of from 1 to 5% by weight.

15 30. A method according to claim 27 wherein the deesterified pectin fraction is recovered from the reaction mixture by adjusting the pH of the mixture to a value in the range of from 3 to 5 by the addition of a base, followed by precipitation of the deesterified pectin fraction in a water-miscible 20 organic solvent or into a homogeneous solution of a water-miscible organic solvent and water and separating the precipitated pectin fraction.

31. A method according to claim 23 wherein the obtained deesterified pectin fraction has a degree of amidation which 25 is in the range of from 5 to 25, including the range of 15 to 25.

32. A method according to claim 23 wherein the ratio between the degree of esterification and the degree of amidation in the resulting deesterified pectin fraction is at least 0.75, 30 such as in the range of 0.75 to 2.00 including the range of 1.0 to 1.5, e.g. in the range of 1.0 to 1.2.

33. A method according to claim 23 wherein the deesterification is carried out in a suspension of a selected high-esterified pectin fraction obtainable by the method of any of 5 claims 1-17, or of a deesterified pectin fraction obtainable by the method of any of claims 23-32 in an aqueous solution comprising a water-miscible organic solvent and ammonia.

34. A method according to claim 33 wherein the amount of pectin dry matter in the suspension is in the range of 10 to 10 30 wt% of the suspension.

35. A pectin fraction obtainable by the method of any of claims 23-34, the fraction having a degree of esterification which is less than 50% and a degree of amidation which is in the range of 0 to 25.

15 36. A pectin fraction according to claim 35 having a degree of esterification which is at the most 45% including at the most 40%.

37. A pectin fraction according to claim 35 or 36 which has a degree of amidation which is in the range of 5 to 25.

20 38. A pectin fraction according to claim 35 wherein the ratio between the degree of esterification and the degree of amidation in the resulting deesterified pectin fraction is at least 0.75, such as in the range of 0.75 to 2.00 including the range of 1.0 to 1.5, e.g. in the range of 1.0 to 1.2.

25 39. Use of a pectin fraction according to any of claims 35-38 in the preparation of a food product.

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